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Maraging steel panels were coated with finishes of paint per MIL-P-23377, vacuum-deposited aluminum, vacuum-deposited cadmium, flame-sprayed aluminum, and electroless nickel, and were exposed to 1200 hours of salt spray and humidity testing. In addition, tests were conducted to determine reaction effects between the paint coating and the HEN-12 propellant.

The vacuum cadmium and paint coatings performed best in protecting the maraging steel panels. The reaction effects between the paint coating and HEN-12 propellant were considered negligible.

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EVALUATION OF COATINGS FOR DRAGON GAS GENERATOR APPLICATION

by

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Maraging steel panels were coated with finishes of paint per MIL-P-23377, vacuum-deposited aluminum, vacuum-deposited cadmium, flame-sprayed aluminum, and electroless nickel, and were exposed to 1200 hours of salt spray and humidity testing. In addition, tests were conducted to determine reaction effects between the paint coating and the HEN-12 propellant.

The vacuum cadmium and paint coatings performed best in protecting the maraging steel panels. The reaction effects between the paint coating and HEN-12 propellant were considered negligible.

ACKNOWLEDGMENT

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1. Introduction

a. Background

Three failures were recorded during DRAGON Gas Generator Design Verification Testing conducted at McDonnell Douglas Astronautics Company (MDAC), Titusville, Florida. The gas generators ruptured at pressures below the design limit load. The rupture was attributed to corrosion in each case. The need for a protective finish for the maraging steel material was evident, but the requirements were restrictive. The finish had to be compatible with nitroglycerine vapors from HEN-12 propellant and virtually smokeless. In addition, its composition had to be such that flare window contamination would not occur. In an October 1970 DF, the DRAGON Project Office requested that the Ground Equipment and Materials Directorate conduct paint and metallic coating tests to assist the DRAGON Project Office and MDAC in selecting an acceptable coating for the gas generator.

b. Purpose

The objective of this evaluation was to show the relative merits of a few selected coatings in protecting maraging steel from corrosion in humid and salt spray environments. The objective also was to show the compatibility relationship of epoxy primer MIL-P-23377 and HEN-12 propellant.

2. Discussion

a. Test Description

Specimens were prepared and evaluated in salt spray and humidity tests. In addition, samples of the MIL-P-23377 paint coating were sent to Picatinny Arsenal for propellant stability test in contact with HEN-12 propellant. Other samples of the paint were sent to the Test and Evaluation Directorate, Redstone Arsenal, for propellant-vapor compatibility tests.

The salt spray test was maintained with a 5-percent salt solution and in accordance with the procedures of MIL-E-5272. The humidity test was maintained at 95 to 100 percent relative humidity between temperatures of 75° and 155°F and cycled in accordance with MIL-E-5272. Both tests were terminated after 1200 hours of exposure.

The propellant stability test was conducted with the MIL-P-23377 paint coating to determine what effects the paint would have on the HEN-12 propellant. The metallic coatings were considered inert. Small

pieces of a coated panel were mixed with the propellant and heated to 194°F for 40 hours. Gas evolution was measured and the increase above the standard amount for the propellant was recorded.

The propellant-vapor compatibility test was an additional check on the MIL-P-23377 paint coating to determine if the nitroglycerine vapors from the propellant would cause excessive softening, loss of adhesion, or other detrimental effects. Test samples, three panels and four canisters, were subjected first to temperatures of 65° and 155°F for five cycles (ten days). One test cycle consisted of 24 hours at 65°F and 24 hours at 155°F. The second phase of the test was a six-weeks soak at 145°F. The third and final phase of the test was a 24-hour temperature shock exposure to 155°F for 4 hours, then -65°F for 4 hours in accordance with Method 503 of MIL-STD-810. During testing, the three panels were in desiccator jars with several sheets of the HEN-12 propellant. The canisters, however, were given a production load of the propellant sticks and sealed. Only the interior surfaces of the canisters were exposed to the propellant vapors. The fourth canister was utilized as a control and only exposed to the temperature cycles.

Visual examination and pencil hardness measurements were made after the 145°F soak phase and at the end of the test.

The hardness of the paint coating was measured by the pencil method. Eagle Turquoise brand pencils of varying hardness with the lead ground flat to full diameter were pushed against the paint film at an angle of 30 degrees. The paint film was tested in this manner with pencils of increasing hardness until the film was penetrated. The hardness number of the pencil penetrating the film represented the paint hardness. The type of impression and penetration damage to the paint film is shown in Figure 1.

b. Test Specimens

Maraging steel panels, 4 by 4 by 0.040 inches, similar to the DRAGON gas generator material, were utilized in the tests. In addition, production canisters of the DRAGON gas generator were added to the vapor compatibility tests. A schedule of test specimens and respective coatings is contained in Table I. All specimens were coated in-house with the exception of the vacuum-aluminum and vacuum-cadmium specimens. These were furnished by MDAC. Cleaning, prior to coating, consisted of degreasing with acetone, treatment with an acid-detergent solution (Oakite 33), and rinsing in water.

Table I. Schedule of Tests, Test Specimens, and Coatings

	Salt Spray Panels	Humidity Panels	Propellant Stability Panels	Vapor Compatibility Specimens	Coating Thickness (in. approx.)
Epoxy Primer MIL-P-23377 Coating	3	3	1-cut into 1/4 × 7/8 pieces	3 panels 4 canisters	0.0015
Vacuum Aluminum Coating	3	2			0.001
Flame-Sprayed Aluminum Coating, "A"	3	3			0.007
Flame-Sprayed Aluminum Coating, "B"	3	3			0.005
Vacuum Cadmium Coating	3	3			0.0005
Electroless Nickel Coating	3	3			0.0005

3. Results

a. Salt Spray Panels

A group photo of the salt spray specimens after 1200 hours of exposure is shown in Figure 2. A closer view of the respective coatings is shown in Figures 3 through 7.

The MIL-P-23377 primer coating performed better than the appearance of the panels suggest. Rust developed at the edges and ran down causing a staining of the panel faces. The edges were not coated with the epoxy primer during normal spraying operations, but were later brush coated with an olive-drab alkyd paint. However, blisters, two on one panel and one on another, did develop in the coating. No rust was found under the blisters. Close examination of the three blisters is

shown in Figure 8. A yellow solution was found under each blister, but the maraging steel substrate was unaffected. The primer contains strontium chromate pigment, which probably formed a chromate solution and prevented rusting. Chromate solutions are noted for their rust inhibiting characteristics.

The specimens with the vacuum aluminum coating developed heavy white corrosion products early in the salt spray test, but did not develop rust until approximately the 650-exposure hour. At the termination point, 1200 hours, two of the panels had developed heavy rust, but the third panel exhibited only white corrosion products.

Both the "A" and "B" panels with flame-sprayed aluminum coating corroded early in the salt spray test; but only the thinner of the two coatings, the "B" panels, began to develop rust near the end of the test.

The vacuum cadmium coating protected the maraging steel panels during the full 1200-hour salt spray exposure without any type of rust development, even at edges and holes. The only action the salt spray seemed to have on the coating was a darkening effect, apparently a thin oxidized layer of cadmium.

The panels with the electroless nickel coating apparently had pin hole defects and developed pitting corrosion in the first 24 hours of testing. Few new spots appeared as the test progressed.

b. <u>Humidity Panels</u>

A group view of the humidity specimens after 1200 hours of exposure is shown in Figure 9. Figures 10 through 16 give a more detailed look at the respective coatings.

The humidity test had no apparent deleterious effect, even at the edges, on the MIL-P-23377 primed panels. The edges of these panels were coated with the olive-drab alkyd paint. However, as shown in Figure 11, tiny blisters were noted after a malfunction in the humidity cabinet. A temperature rise to 210°F was recorded after approximately a week of exposure. It took approximately 1-1/2 hours for the chamber temperature to return to 155°F, the normal temperature of the cycle at the time of excursion. Close examination after the full 1200-hours exposure indicated that the blisters contained no moisture nor was corrosion apparent under them.

The vacuum aluminized panels developed light oxidation of the aluminum coating, a few small blisters, and light rust at holes and edges in the humidity test. Figure 13 shows a close-up of the rust and blister formation. The blisters were first observed after the high temperature malfunction in the test cabinet.

The panels with the flame-sprayed aluminum coating, both the "A" and "B" thicknesses, developed rust early in the humidity test. However, the "B" thickness panels (thinner coating) appeared to have more rust at the end of the test.

The vacuum cadmium coated panels darkened considerably from the humidity test, but did not show any rust development.

The electroless nickel-coated specimens developed pitting corrosion in the humidity test. This condition developed in the initial stages of the test and occurred primarily on only one of the three panels.

c. Propellant Stability Test Specimens

Representative pieces of the control and test samples that underwent the propellant stability test are shown in Figure 17. The evolved gas caused by the MIL-P-23377 paint coating was measured to be 0.03 millimeters, well below the acceptable limit of 5 millimeters. The propellant caused the paint coating to soften and darken as shown in Figure 17, but it did not affect paint integrity. Pencil hardness measurements indicated the paint softened from 2H hardness to H hardness, which is not considered significant or detrimental. Pencil hardness measurements for the paint are contained in Table II.

d. Propellant-Vapor Compatibility Specimens

The MIL-P-23377 coating on the specimens in the vapor compatibility test performed without major discrepancy up through the 6-weeks soak at 145°F. Only minor softening and discoloration occurred. Pencil hardness measurements indicated a change from 2H to F. This was a decrease of two hardness values but the drop did not cause any significant change in the paint integrity. The paint did not lose adhesion, blister, nor was it easily scratched with the fingernail. However, after the last phase of the test, the 24-hour temperature shock test, the canister samples including the control, developed coating failure. At the edges of the burst diaphragms in several spots on the interior of the canisters, the paint lost adhesion and peeled. Figures 18 through 20 show this type damage. The damage was more severe in the control canister than in the test items. It appears the softening effects induced by the propellant vapors gave the paint more resistance to the damaging stresses. A plausible explanation for the late failure in the paint is not clear, but it is possible that the expansion differences between the maraging steel canister and aluminum burst diaphragm over stressed the paint.

Table II. Pencil Hardness Measurements on MIL-P-23377 Coating

Hardness Values*	Remarks
2H	After 72 hours air dry
3H	After 72 hours air dry and 24 hours bake at 145°F. Measurement taken at room temperature.
H	After 40 hours contact with HEN-12 Propellant at 194°F. Measurements taken at room temperature.
F	After exposure to HEN-12 vapors for ten days at alternating temperatures of 65°F and 155°F, then 145°F for six weeks. Measurements taken at room temperature.

*Pencil Grade Chart:

$$\underbrace{9H\ \ 8H\ \ 7H\ \ 6H\ \ 5H\ \ 4H}_{\text{Hard}}\quad \underbrace{3H\ \ 2H\ \ H\ \ F\ \ HB\ \ B}_{\text{Medium}}\quad \underbrace{2B\ \ 3B\ \ 4B\ \ 5B\ \ 6B\ \ 7B}_{\text{Soft}}$$

4. Conclusions and Recommendations

a. Conclusions

The vacuum cadmium coating is concluded to be the best coating of the finishes tested. The coating protected the maraging steel panels under the enviromental conditions.

The MIL-P-23377 primer coating is concluded to be an effective coating for the conditions imposed. The blister damage that occurred in the humidity test is considered a heat-imposed discrepancy and should be discounted. The slight blister development in salt spray does suggest that with a single coat of paint some pin holes could form.

The paint failure that occurred inside the canister specimens in the temperature shock phase of the propellant-vapor compatibility tests can be disregarded. Sudden dimensional changes by expansion and contraction of dissimilar metals can bring about a coating failure as was experienced. However, the shock conditions of the test were much more harsh than will be encountered in the field.

The vacuum aluminum, flame-sprayed aluminum, and electroless nickel coatings allowed the maraging steel substitutes to rust. Therefore, their protection rating is concluded to be below that of the cadmium and paint coatings, but their order relative to each other was approximately the same.

It is concluded that the MIL-P-23377 coating has a negligible effect on the stability of the HEN-12 propellant. Accordingly, it is concluded that the HEN-12 propellant has a negligible effect on the paint. The softening and discoloring effects on the paint from the propellant vapors were not considered detrimental to the protection of the canister.

b. Recommendations

The MIL-P-23377 primer coating is recommended as the protective finish for the DRAGON gas generator. However, two coats of the material instead of one, are recommended to add more reliability against pin hole development. This would amount to a paint film build-up of 2 mils nominally.

The vacuum cadmium coating is not considered to be sufficiently superior to the paint to warrant the higher coating cost.

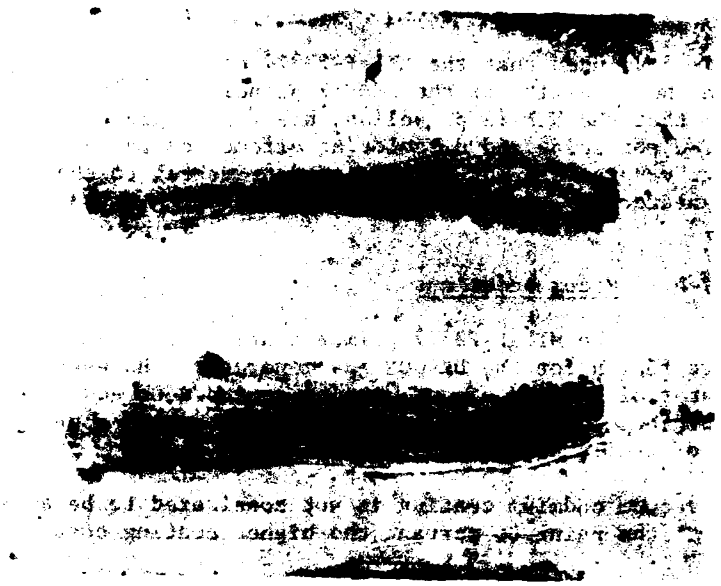

Figure 1. Pencil Hardness Measurements. (The photo is typical of pencil hardness impressions made on the MIL-P-23377 epoxy paint coating. These impressions are on a control sample of the paint that had been air dried for 72 hours. Penetration of the film occurred with a 2H lead. A complete summary of hardness values for the paint is contained in Table II.)

Figure 2. Salt Spray Panels. (The maraging steel panels, three of each coating, show 1200 hours of salt spray testing. The coatings in the top row, left to right, are electroless nickel and epoxy primer MIL-P-23377. The coatings in the middle row are vacuum cadmium and flame-sprayed aluminum "A". The coatings in the bottom row are flame-sprayed aluminum "B", and vacuum aluminum.)

Figure 3. MIL-P-23377 Coating, Salt Spray Exposure. (The photo shows the MIL-P-23377 coating on maraging steel panels after 1200 hours of salt spray exposure. The rust is coming from edges which were not protected with the epoxy material but with an olive-drab alkyd paint. The only failure in the coating is three blisters; one on the right panel and two on the left panel. See Figure 8 for observations about the blisters.)

Figure 4. Vacuum Aluminum Coating, Salt Spray Exposure. (The photo shows vacuum-aluminized maraging steel panels after 1200 hours of salt spray exposure. Two of the three panels developed heavy rust.)

Figure 5. Flame-Sprayed Aluminum Coating, Salt Spray Exposure. (The photo shows the flame-sprayed aluminum samples after 1200 hours of salt spray exposure. The top row is the "A" thickness and the bottom row the "B" thickness. Although all the panels are showing considerable white corrosion, only the "B" panels, the thinner coating of the two, are showing traces of rust.)

Figure 6. Vacuum Cadmium Coating, Salt Spray Exposure. (The photo shows the vacuum-cadmium maraging steel panels after 1200 hours of salt spray exposure. The coating developed only a light oxide layer and darkening effect from the test.)

Figure 7. Electroless Nickel Coating, Salt Spray Exposure. (The photo shows the electroless nickel-coated maraging steel panels after 1200 hours of salt spray exposure. The pits shown in the coating developed early in the test.)

Figure 8. Paint Blister Damage. (The photo shows a typical condition of the maraging steel panels under the blister damage in the MIL-P-23377 coating after 1200 hours of salt spray exposure. The three blisters contained a yellow solution, apparently a chromate solution, which prevented the development of rust.)

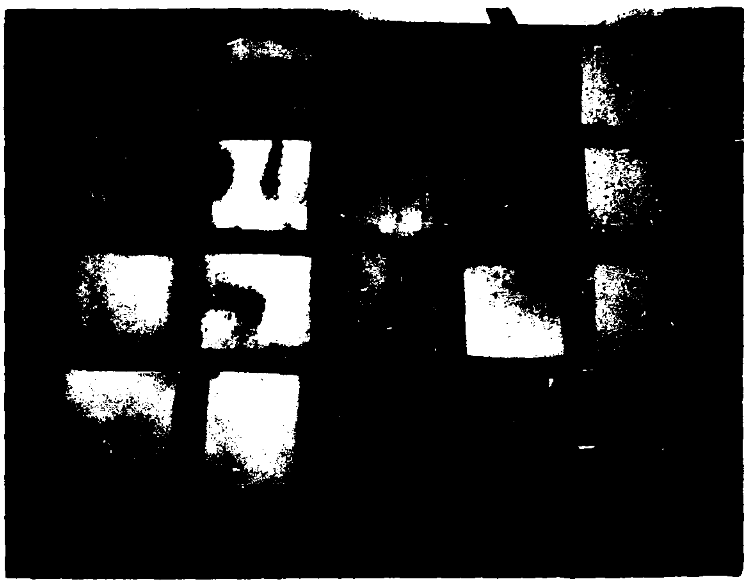

Figure 9. Humidity Panels. (The maraging steel panels show 1200 hours of humidity testing. The coatings, three specimens each, from right to left are epoxy primer MIL-P-23377, electroless nickel, vacuum cadmium, flame-sprayed aluminum coating "A", and flame-sprayed aluminum coating "B". The two panels at the bottom, across, are vacuum aluminum coated.)

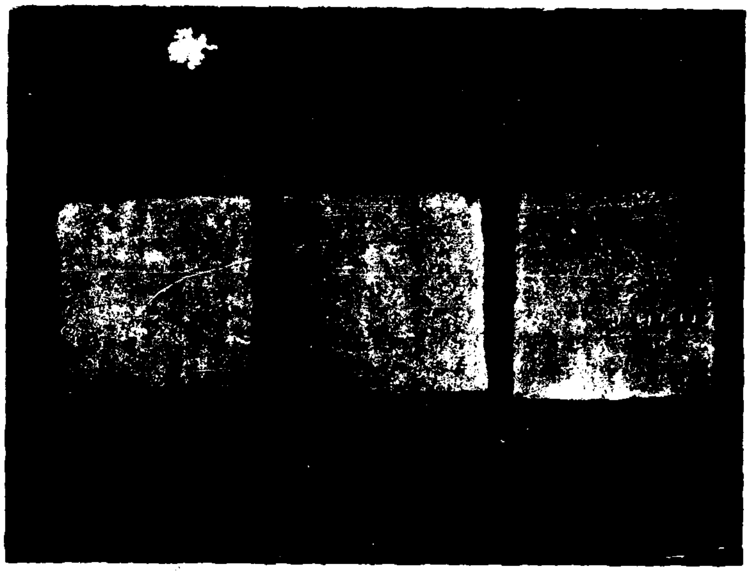

Figure 10. MIL-P-23377 Coating, Humidity Exposure. (The photo shows maraging steel specimens with the MIL-P-23377 coating after 1200 hours of humidity exposure. The exposure did not produce corrosion even at the edges which were coated with an alkyd paint. However, blister damage did occur (Figure 11), but was believed to be caused by a temperature excursion in the test equipment.)

Figure 11. MIL-P-23377 Coating, Blister Damage. (The photo shows blister damage that occurred in the MIL-P-23377 coating after approximately one week of humidity testing. This condition was noted after the test equipment recorded a temperature rise to 210°F. The blisters did not appear to increase in size or number through the full 1200 hours of humidity exposure. The blisters were found void of moisture and corrosion.)

Figure 12. Vacuum Aluminum Coating, Humidity Exposure. (The photo shows vacuum-aluminum maraging steel panels after 1200 hours of humidity exposure. Only two panels were available for the humidity test. Three panels were utilized for all the other coatings. Only light oxidation of the aluminum coating developed on the faces of the panels; however, traces of rust developed at the edges and at the drilled holes. A blister phenomenon also developed as illustrated in Figure 13.)

Figure 13. Blister and Rust Damage. (The photo shows the vacuum-aluminized panels after 1200 hours of humidity exposure. The blister phenomenon, which occurred over the entire face of the panels but more pronounced at the edges, was first noticed after approximately a week of exposure when the test equipment registered a temperature rise to 210°F. Rust occurred primarily at holes but traces can also be seen at the bottom edge of the right panel.)

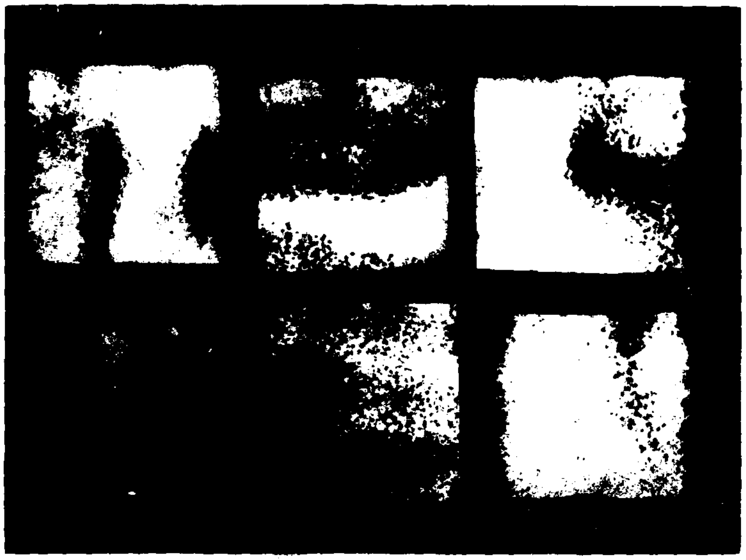

Figure 14. Flame-Sprayed Aluminum Coating, Humidity Exposure. (The photo shows the flame-sprayed aluminum coated panels after 1200 hours of humidity exposure. The top row is the "A" thickness and the bottom row is the "B" thickness. The "B" panels, the thinner coating of the two, appear to show more rust. These conditions developed early in the test and did not change much during the progress of the test.)

Figure 15. Vacuum Cadmium Coating, Humidity Exposure. (The photo shows the vacuum-cadmium coated maraging steel panels after 1200 hours of humidity exposure. The primary change in the coating was a darkening effect.)

Figure 16. Electroless Nickel Coating, Humidity Exposure.
(The photo shows the electroless nickel-coated panels after 1200 hours of humidity exposure. The pitting conditions developed early in the test and showed little change as the exposure increased. The coating on only one panel was critically defective.)

Figure 17. Propellant Stability Test Specimens. (The photo shows representative samples of the 1/4- by 7/8-inch pieces that were mixed with HEN-12 propellant at Picatinny Arsenal and subjected to a 194°F bake for 40 hours. The two pieces on the right are representative of the control samples and did not come in contact with the propellant. The two pieces on the left are from the test samples. The paint, MIL-P-23377 epoxy primer, had negligible effect on the propellant. However, the propellant discolored and softened the paint, but not to the extent considered damaging to the performance of the coating.)

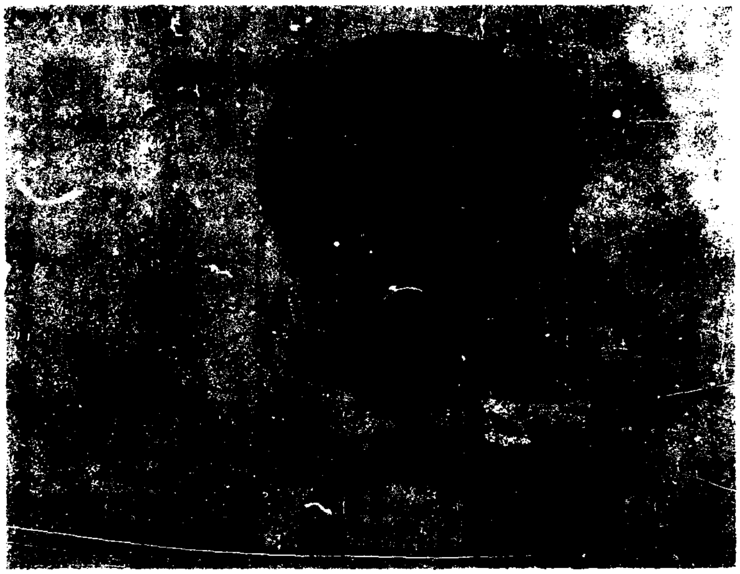

Figure 18. Control Specimen, DRAGON Canister. (The primed gas generator canister shown was used as a control specimen in the propellant-vapor compatibility tests, and experienced only the temperature exposures of the tests. Paint failure occurred inside the canister at the burst diaphragm after the temperature shock phase of the tests. A mirror was placed in the canister to obtain photographic coverage of the damage. Figures 19 and 20 show the relative extent of the damage in the control canister and test canisters.)

Figure 19. Control Canister, Paint Failure. (The photo shows adhesive failure in the MIL-P-23377 coating of the control specimen after the temperature shock phase of the propellant-vapor compatibility tests.)

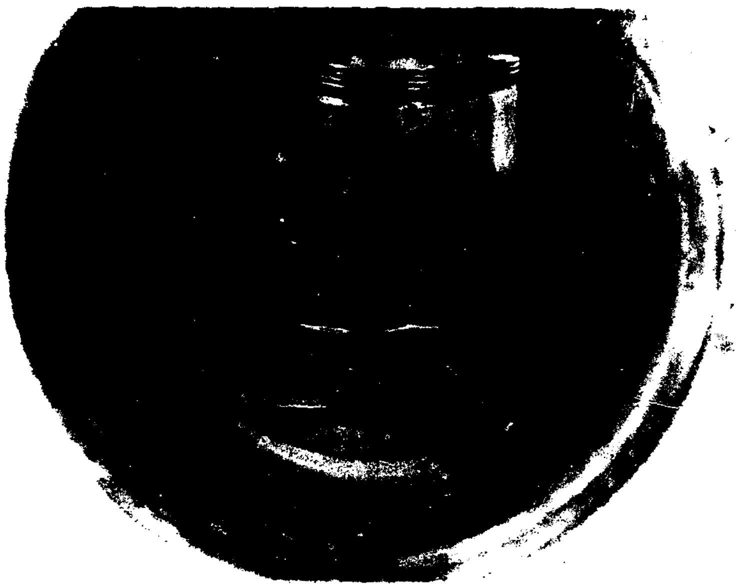

Figure 20. Test Canister, Paint Failure. (The photo shows typical adhesion failure in the MIL-P-23377 coating of the test canisters after the temperature shock phase of the propellant-vapor compatibility tests. The softening effect from the propellant vapors apparently decreased the severity of the damage.)